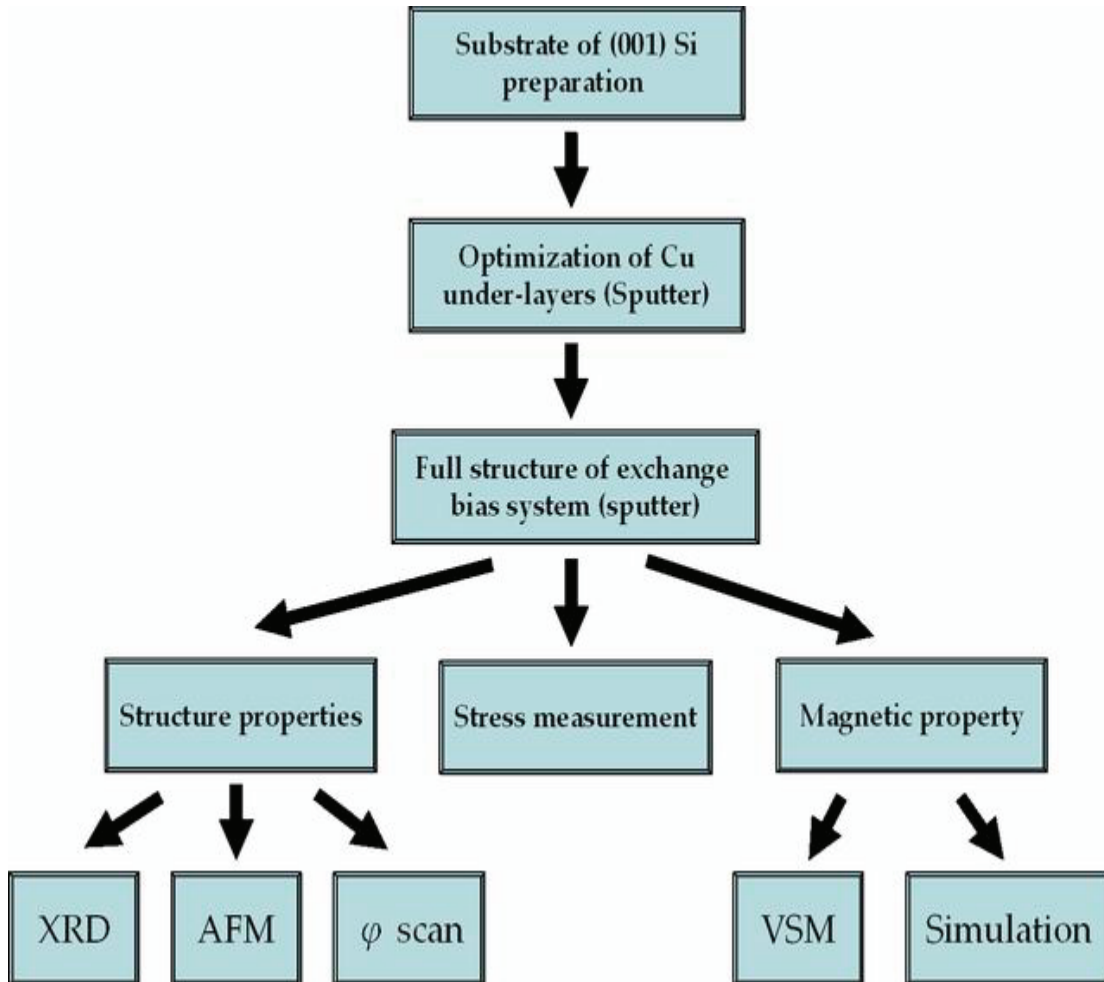


## Chapter 3 Experiment Procedure

### 3-1 Experiment Flow Chart



## 3-2 High Vacuum Sputtering System

In this thesis, the FM/AFM bilayers were prepared by using DC sputtering system. This system, as shown in Fig. 3.1, has five sputtering guns. The sputtering system possesses two main parts: one is the growth chamber, another is a loading chamber. The pumping system contains a mechanic pump, a turbo pump, and a cryo pump. The best pressure in this system can be down to  $4\sim7\times10^{-7}$  Torr.

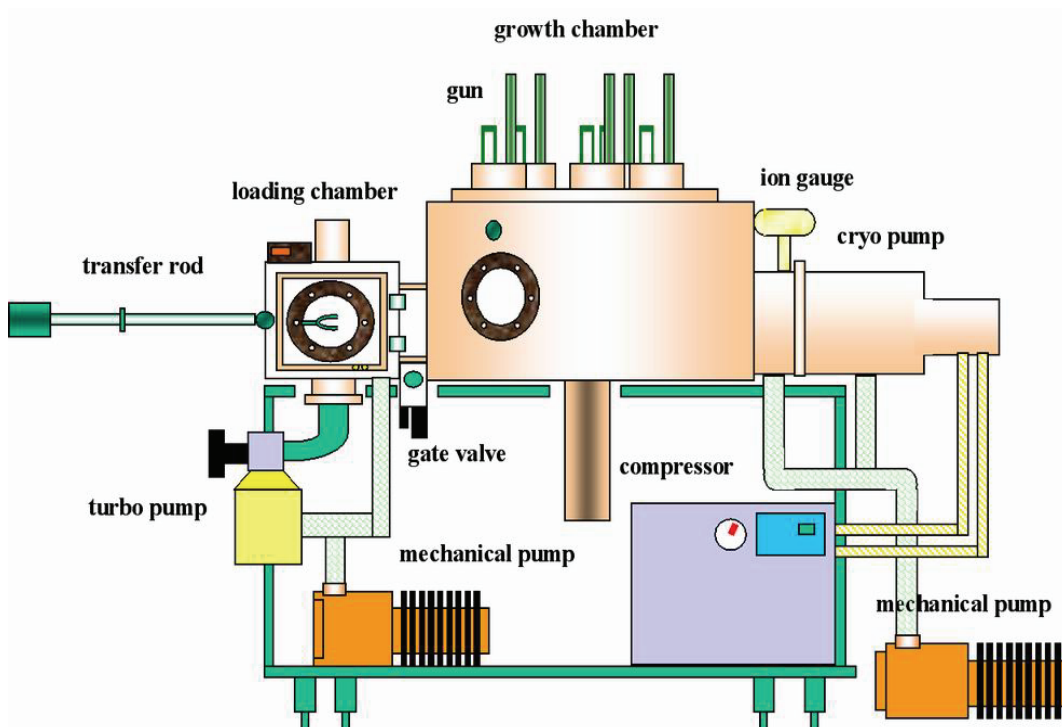


Fig. 3.1 Sputtering system

### 3.3 Analysis Technique

#### 3.3.1 Structure Measurements

##### 3.3.1.a X-Ray Diffraction (XRD)

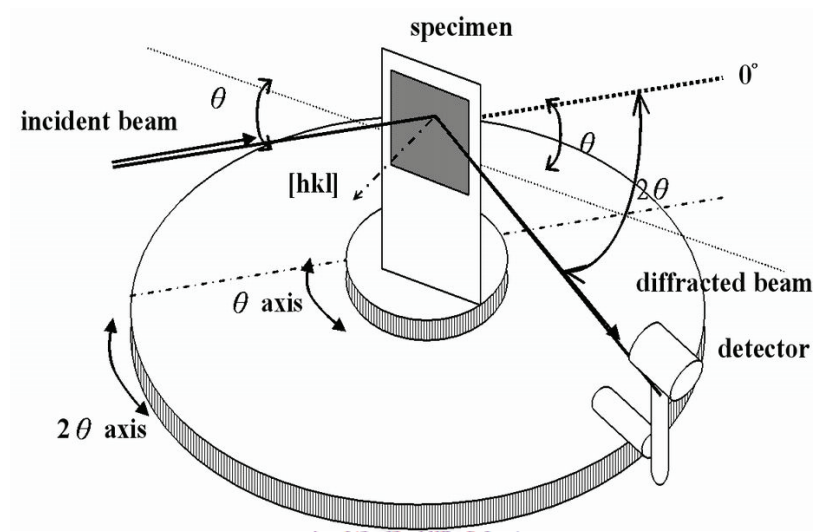


Fig. 3.2 XRD spectrometer.

The essential features of XRD instrument is shown in Fig. 3.2. The rotating axis of specimen and detector are defined as  $\theta$  and  $2\theta$  axis respectively. The detector can be rotated about sample and set at any desired angular position. The crystal is usually cut or cleaved so that a particular set of reflecting planes of known spacing is parallel to its surface, as suggested by the drawing. In use, the specimen is positioned so that its reflecting planes make some angle  $\theta$  with the incident beam, and the detector is set at the corresponding angle  $2\theta$ . The intensity of the diffracted beam is then measured and its wavelength is calculated from the Bragg law “ $2d\sin\theta=n\lambda$ .” This procedure could be repeated for various angles  $\theta$ . The directions in

which a beam of given wavelength is diffracted by a given set of lattice planes are dependent on the crystal system to which the crystal belongs and its lattice parameters. It means significantly that it is possible to determine about an unknown crystal by measurements of the directions of diffracted beams are the shape and size of the unit cell. In addition, the intensities of diffracted beams are determined by the positions of atoms within the unit cell.

### 3.3.1.b $\phi$ Scan

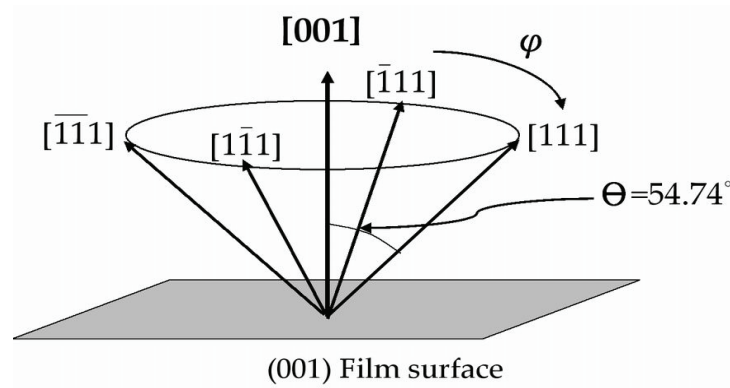


Fig. 3.3 schematic illustration of  $\phi$  scan.

To verify the in-plane orientation and the epitaxial relationships between layers, asymmetric in-plane  $\phi$  scan were performed in a four-circle X-ray diffractometer. Asymmetric scans are used to measure peaks from planes that are not parallel to the film surface. The principle of the  $\phi$  scan is schematically illustrated in Fig. 3.3. As shown in the figure, if the film is cubic (001) textured with random in-plane orientations, all normal directions of equivalent  $\{111\}$  type planes are uniformly distributed on the surface of the cone with the angle of  $54.74^\circ$  from the  $[001]$  film normal; on the other

hand, if the film is cubic (001) epitaxial with a single in-plane orientation, only four {111} normal directions lie on the surface of the cone due to the 4-fold symmetry of {001} planes. Consequently, if we tilt the sample to an off-axis orientation, e.g., the  $\langle 111 \rangle$  direction,  $54.74^\circ$  away from the film normal  $\langle 001 \rangle$  direction and fix the  $2\theta$  at  $40.5^\circ$ , corresponding to the d-spacing of IrMn {111} planes, the diffraction scans by rotating the sample along the film normal direction should enable us to distinguish epitaxial films from textured films. This procedure is defined as a  $\phi$  scan. For textured films, the intensity of the diffracted beam is uniformly distributed over the entire range of  $\phi$  angle, whereas for epitaxial films with a single in-plane orientation, only four strong peaks are visible.

### 3.3.1.c Atomic Force Microscopy (AFM)

The atomic force microscope (AFM) is a very high-resolution type of scanning probe microscope. In this study, we get the information of surface profile and film thickness by using tapping mode of the AFM, as illustrated in Fig. 3.4. An atomically sharp tip is scanned over a surface with feedback mechanisms that enable the piezo-electric scanners to maintain the tip at a constant force to obtain height information above the sample surface. Tips are typically made from  $\text{Si}_3\text{N}_4$  or Silicon, and extended down from the end of a cantilever. The nanoscope AFM head employs an optical detection system in which the tip is attached to the underside of a reflective cantilever. A diode laser is focused onto the back of a reflective cantilever. As the tip scans the surface of the sample, moving up and down with the contour of the surface, the laser beam is deflected off the attached cantilever into a dual element photodiode. The photo-detector measures the difference in light

intensities between the upper and lower photo-detectors, and then converts to voltage. Feedback from the photodiode different signal, through software control from the computer, enables the tip to maintain either a constant force or constant height above the sample.

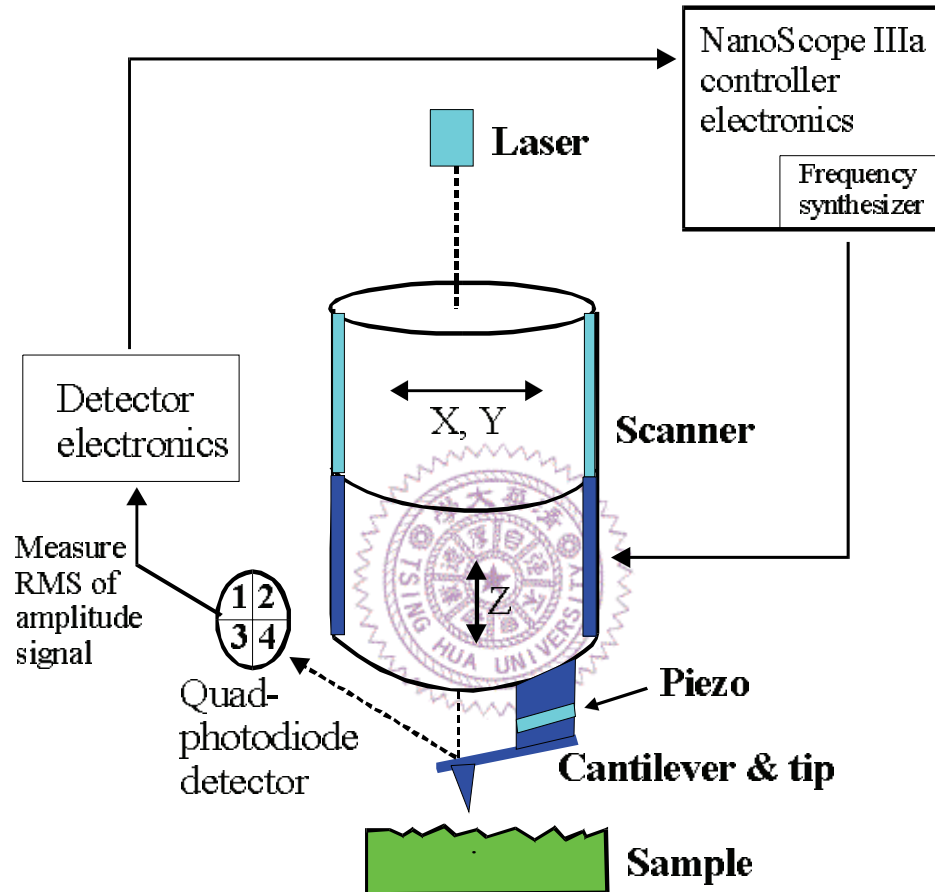


Fig. 3.4 schematic illustration of AFM

### 3.3.2 Magnetic properties Measurements

#### Vibrating Sample Magnetometer (VSM)

The measurement method was proposed by Foner. The principle of VSM is vibrating the sample near the coil, which would induce the variation of magnetic flux. The sample can be in the useful shape of rod or sheet and is mounted on one end of the test rod. The other end of the test rod is connected to loudspeaker. In the beginning with measuring, the testing rod vibrates together with sample with 80Hz frequency. The direction of vibration is perpendicular to the magnetic field. An induced electromotive force would be occurred in the coils due to the variation of magnetic flux though the detection coils. With a reference sample (the saturation moment  $M_s$  is known) fixed at the testing rod vibrating together with the measured sample, there is also an induced electromotive force formed in the reference coils. Therefore, we can obtain the magnetic moment of measured sample by comparing the induced electromotive force in the detection coils with reference coils. The scheme of VSM is illustrated in Fig. 3.5(a).

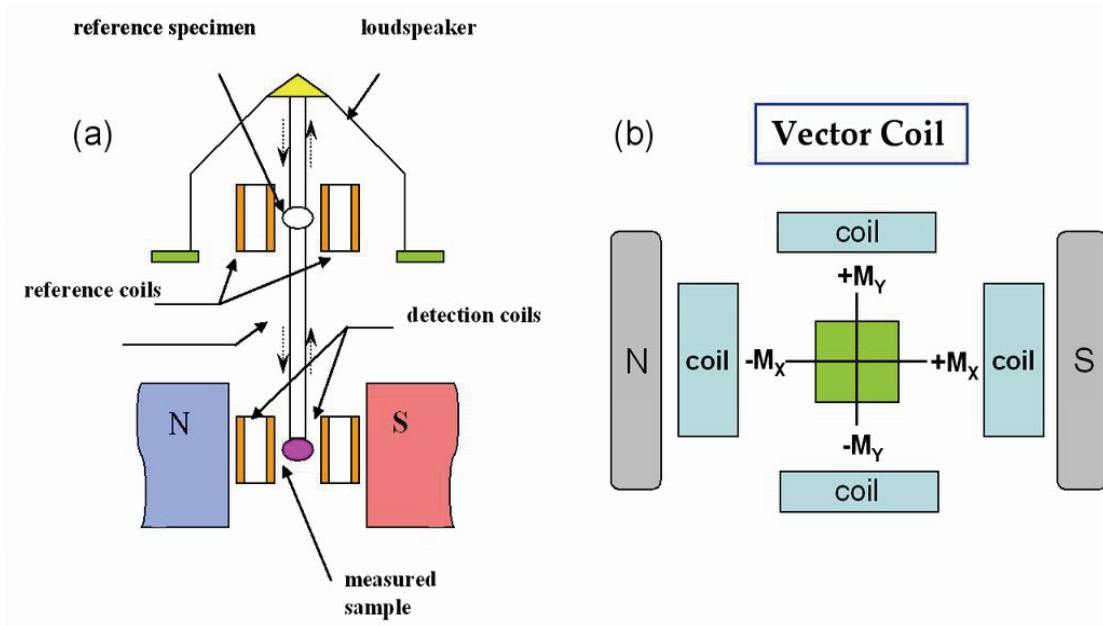


Fig 3.5 (a) Schematic illustration of VSM. (b) Schematic illustration of vector coil.

In our experiments, the hysteresis loops measured from a vector coil, seen in the Fig. 3.5(b), a pick-up coil locating in the orthogonal direction (y-direction) to the applied field direction (x-direction), provide understanding of magnetization reversal in the (001) IrMn/CoFe system.



### 3.3.3 Stress measurement

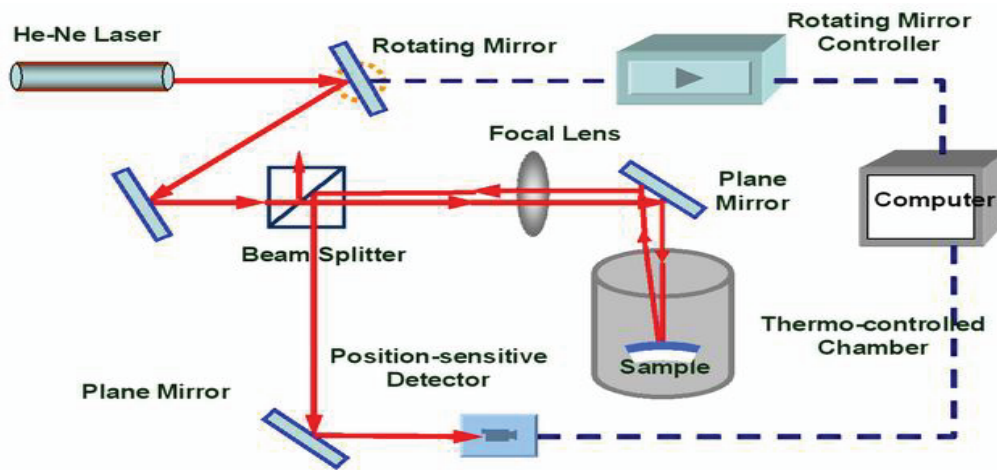


Fig. 3.6 Curvature measurement.

The curvature measurement system, as described in Fig. 3.6, combines He-Ne laser with rotating mirror, and can measure the curvature with raising temperature process in short time instead of moving samples. The wavelength and power of He-Ne laser are 632.8 nm and 7 mW, respectively. Laser beam would be reflected by rotating mirror which is dominated by rotating mirror controller (General Scanning Inc. CX-600) and able to alter scan rate and region. Subsequently, the laser beam would forward to and reflected by plane mirror, and then passes through the beam splitter as well as divided into two directions of beams. One is reflected into air while the other is lead to focal lens and reflected onto the sample in thermo-controlled chamber. According to various curvatures, the reflected beam from sample would get back along same trace to beam splitter and be introduced into position-sensitive detector. The result of curvature would be obtained from this system by linking to the computer.